National Bureau of Standards

July 1959
Volume 43
Number 7

Technical News Bulletin

Geomagnetic Effects of Nuclear Explosions

THE BUREAU has been analyzing the changes that occurred in the earth's magnetic field when two nuclear bombs were exploded by this country at high altitudes. Results indicate that these nighttime explosions ionized the upper atmosphere at a distance of 2,000 km to nearly daytime intensity, resulting in electric current flows that temporarily altered the geomagnetic field. The analysis was carried out by A. G. McNish.

The effects were recorded at the regular magnetic observatories operated at Honolulu, Hawaii, by the Coast and Geodetic Survey, and at Apia, Samoa, by the New Zealand Government, as well as at three special International Geophysical Year stations on Palmyra Island, Fanning Island, and Jarvis Island. The data were sent to the Bureau for study because of its interest in ionospheric phenomena and their effects on radio propagation.

Up to a distance of about 2,500 km from Johnston Island, the firing point, the magnetic field changes are ascribed to primary and secondary gamma radiation from the explosions. The effects at Apia, at a distance of 3,400 km, are attributed to charged particles from the detonation traveling along the lines of the earth's magnetic field toward the magnetic conjugate point just northwest of Apia, where artificial aurora were sighted on both occasions. Stations distant 4,000 km or more from the explosions exhibited no distinguishable effect.

The bombs were fired August 1, and August 12, 1958, near midnight, Hawaiian time. Special rapid-run magnetographs operating at both Honolulu and Apia reveal that the effects began simultaneously at these two stations to within a second or two. Analysis of the results permits calculation of probable heights at which the explosions were set off. The lower of the two, as calculated, was between 35 and 50 km, and the other about 20 km or more higher.

The tests provided an opportunity to confirm certain geomagnetic theories dealing with the quiet diurnal variations and magnetic storms. The Stewart-Schuster theory of the diurnal variations in terrestrial magnetism assumes the existence of two systems of electromotive forces in the atmosphere. These systems result from the tidal oscillation of the atmosphere across lines of force of the earth's permanent magnetic field; one system occurs in the daylight hemisphere and the other in the dark hemisphere. Because of the ionizing action of the sun's illumination, electric currents flow in the daylight hemisphere, but are virtually nonexistent at night when the upper atmosphere has very low conductivity.

Explosion of the weapons produced artificial ionization permitting currents to be driven by electromotive forces already present. The pattern of the resulting current system, which produced the magnetic effects, closely resembles the pattern of the current system regu-

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DETROIT

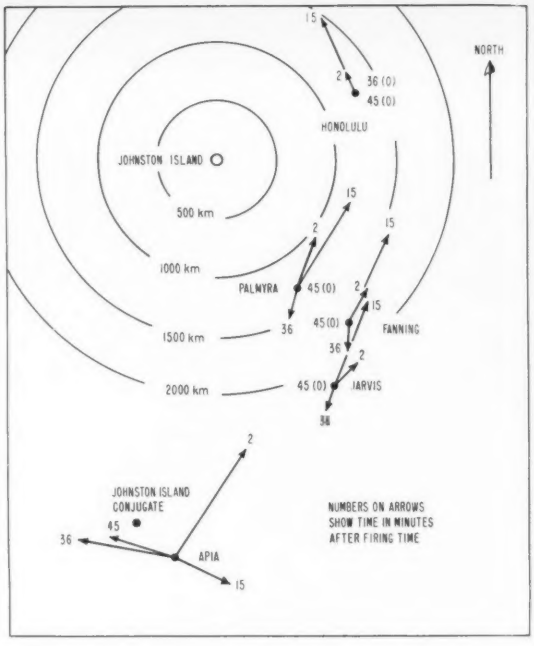
High-altitude nuclear explosions ionized the upper atmosphere to almost daytime intensity, thus altering the earth's magnetic field. Map shows the current sheet vectors at times immediately after an explosion.

larly flowing during the daylight hours. These artificially produced currents persisted for a little over half an hour after the explosion. The magnetic effects at Honolulu, Palmyra, Fanning, and Jarvis were nearly as large as if the lower layers of the ionosphere had been temporarily ionized to daytime intensity.

The magnetic field changes observed at Apia do not fit into this current system, but are due to local effects at the magnetic conjugate point. These effects persisted for well over an hour. The field changes exhibit the erratic fluctuations commonly experienced by stations in high latitudes where auroral particles impinge on the atmosphere.

The electromotive forces generating the currents in the neighborhood of Apia may have had two possible causes. One of these is the atmospheric turbulence resulting from the heating effect of the influx of charged particles. The other possibility is that a magnetic field was propagated from the explosion along the earth's magnetic lines of force to the conjugate point of explosion on the opposite side of the geomagnetic equator.

These are the only such magnetic effects which have so far been reported from the explosions of nuclear devices, except for a small effect at the time of some British nuclear tests conducted at Christmas Island. This effect was observed at Fanning Island, 200 km distant, and at Jarvis Island, 300 km distant. Because of the small size and limited extent of these effects, it is believed that the British explosions took place much lower in the atmosphere than the two high-altitude explosions set off by the United States in August 1958.



Both United States explosions were followed immediately by blackout of high-frequency radio circuits traversing the area of the Pacific Ocean near Johnston Island, according to reports received from commercial circuits operating between Australia and New Zealand and the west coast of North America. The blackouts lasted several hours and were followed by impaired radio conditions for several days.

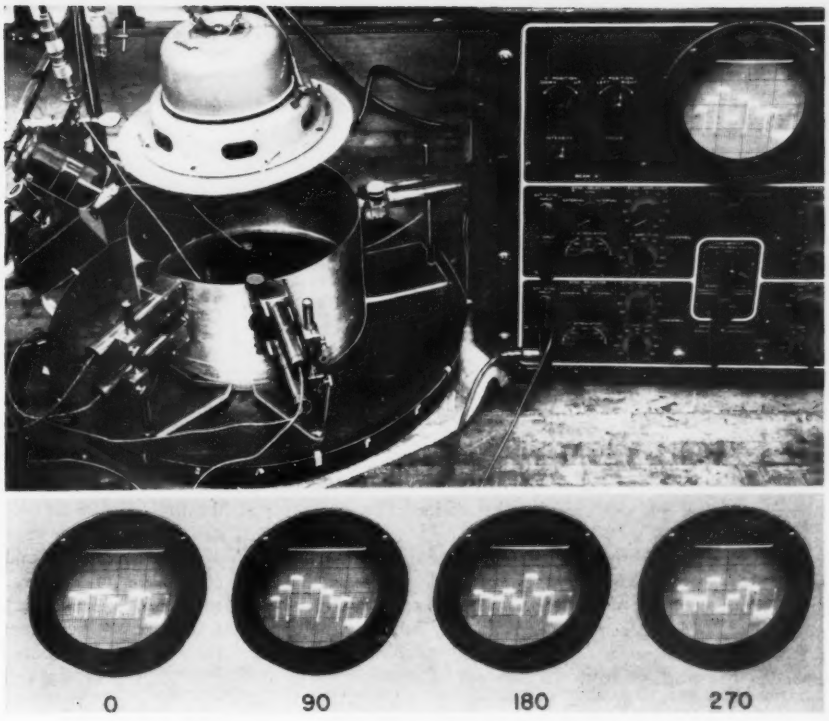
A STROBOSCOPIC VIBRATION ANALYZER

for aircraft and missile structures

A TIMESAVING vibration analyzer has been developed by S. Edelman and others of the Bureau.[1] This analyzer can be used for determining vibration characteristics of bodies such as aircraft and missile structures. The instrument uses the principle of a stroboscope, and enables an operator to quickly explore the vibration patterns in a complex structure. Early in an analysis, therefore, an operator can determine whether his vibration pickups have been located so as to give him the information he needs.

The stroboscopic vibration analyzer was developed for use in calibrating vibration pickups, where vibrations other than longitudinal were unwanted. By using several small pickups on various parts of the shaker, it was possible to eliminate transverse or flexural vibrations. This work on vibration is part of a long-term program aimed at providing additional information on the performance of vibration pickups.

A stroboscope is a convenient instrument for determining the resonant frequencies of bodies vibrating at low frequencies. However, when higher frequencies are examined, the duration of the light pulses becomes shorter, putting a limit on the use of a stroboscope for visual inspection for resonant points. There is an additional limit to the usefulness of a stroboscope in that the vibrating body must be observed. For an examination of very small, even microscopic, movements or parts, this examination becomes difficult. When the movement is submicroscopic, some other method must be used. It is in this area that the vibration analyzer is a useful tool.



Stroboscopic analyzer in operation. Bell-shaped loudspeaker vibrates the cylinder below it. On the displays, horizontal traces show relative amplitude of signals from the analyzer pickups mounted next to the cylinder wall. For changes in phase of the beat frequency, the patterns vary as shown in the bottom row.

With a stroboscope, the periodic changes of position of a given body are displayed in apparent slow motion, or are stopped at some desired phase of the vibration cycle. With the stroboscopic vibration analyzer, the same effect is produced by processing signals from transducers or vibration pickups in such a way as to display the pattern of motion on an oscilloscope. Thus the motion of the body itself need not be observed.

In the analyzer, the motions of a body are first converted to electrical impulses by the pickups, and the resulting signals are sent to a mixer which adds a reference signal slightly lower than the frequency at which the body is vibrated. Next, the difference signals are separated by a low-pass filter or a square-law detector. When the outputs from this process are displayed on an indicator, such as an oscilloscope, the vibration pattern can be readily seen for a given phase. As the body vibration frequency is changed, the reference signal changes a corresponding amount, so that the difference signal remains at the same frequency.

To make a bar-type trace on the oscilloscope instead of a sine wave, a transistor was used as an emitter follower and d-c rectifier. An output transistor was used to invert the negative signals.

By varying the frequency of the vibration of the body, and the positions of the pickups on the vibrating body, a complete analysis can be made of the vibration characteristics of the body.

Flashing lights can be used for the display, and when properly positioned in space, they can give a three-dimensional representation of vibration. A recorder can be connected to the output channels. When a recorder of limited dynamic response is used, the difference frequency can be chosen to suit the recorder.

A voltmeter can be used to read the absolute value of the motion, if desired. To do this, the signal from a given pickup is fed into the voltmeter from a T-connector at the input to its mixer channel. However, the vibration analyzer is more useful for exploring than for routine measuring.

The analyzer is most useful in helping to visualize the varied motion patterns of a complex structure, or in determining the interactions among the varied motions. The usefulness of the instrument depends to some extent on the ingenuity of the user. For example, the motion to be studied need not be controlled. The signal from one pickup can be used as a reference, and applied to each mixer channel by way of the phase shifter.

This device should simplify gathering of data from any periodic process in which the amplitude and phase relations of changes are important. The characteristics of the pickups are the only limits to the vibration amplitudes that can be studied by this method; and by simply changing the oscillator, frequencies of any range can be analyzed.

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[1] *For further technical details, see* A stroboscopic vibration analyzer, *by S. Edelman, R. Brooks, S. Saito, E. Jones, and E. R. Smith, J. Research NBS (to be published).*

SECRETARY OF COMMERCE SPEAKS ON METRIC SYSTEM

Excerpts of remarks by
Lewis L. Strauss, Secretary of Commerce
before the American Physical Society
Washington, D.C.
May 1, 1959

I HAVE long been convinced that ultimately the United States must shift to the metric system. Outside of our Anglo-Saxon culture, practically every nation has made this shift during the past 150 years or so. Due to our delay in taking action and due to the complexity of our industrial system, this change will be more difficult for the United States than for other countries, but when achieved it will also be more useful.

The advantages of the metric system would be many in addition to ease of calculation and conversion. Bookkeeping and computational requirements would be reduced. Our international trade, our buying, and our selling, and many of our international aid and security programs would be facilitated. The metrological divorce between our science, research and development, and our industrial programs would be overcome. The more computer techniques come into significant use the greater will become our need for an efficient system of measurement. The conquest of space will be facilitated if we first make up our minds to use the one measuring system which has the greatest potential for growth and accuracy.

Actually at the present time, we are using both systems of measurement. Slide rules and desk calculators are metric in nature. The decimal point with which we adorned the English system is an eloquent witness to the indispensability of the inevitable metric measurement. We switched from pounds and shillings to dollars and cents. In the chemical and electronics industries, the metric system is in widespread use. Our electrical measurements are all based on the MKS system. Perhaps it is significant to note that the pharmaceutical industry made its own decision to go over to the metric system in the past few years. And, lest we forget, it has been medical practice in these United States to weigh all new-born babies in grams.

Thus, the metric system is all around us. The problem, therefore, is not to make a revolution in measurement but to direct and accelerate the evolution which has been taking place. It is a matter of becoming conscious of this evolution and of giving it an assist. It is a matter of using the metric system more and more and the English system less and less.

Naturally this evolutionary process will take some time. We still have very considerable amounts invested in the English system of measurement. Our culture has long been entrenched in yards, pounds, and gallons, and a reeducation will be necessary. Textbooks will require revision and instruments will require recalibration or replacement. The changes in our machine tools and in much of our equipment—containers for example—will be most difficult and costly. The switch from inches to centimeters in industry must be accomplished with minimal disruption, but I do not think it will take years upon years.

We simply cannot remain static but must recognize the conditions of our time and meet rather than be forced into the inevitable and logical. The time has come when the Government should do something affirmative about a proposal which scientists have been reiterating since the days of Lord Kelvin.

Recently a committee to consider the problems of shifting to the metric system was established by the American Association for the Advancement of Science after it had approved in principle the general adoption of the metric system of weights and measurements.

Our country had a number of opportunities earlier in its history to adopt the metric system and in truth it would have been much easier at those earlier dates. Each time we delayed, the problem of a change became more difficult because we were becoming more industrially entrenched in our commitments to the English system. The cost of the changeover has increased and an orderly transition has become more difficult. What are the considerations that appear as though they will compel us to metricize ourselves?

They are of two kinds, scientific and economic. The advances in research in the past 20 years lead but to one conclusion as far as measurement is concerned. We can no longer afford the luxury of national measurement systems which differ from those of the people with whom we want to trade. Furthermore, the metric system lends itself most easily to providing an interrelated network covering all scientific fields. The fact that science is on the metric system and that civil engineering and industry by and large are on the English system makes for a great deal of unnecessary confusion and difficulty.

There are two compelling economic reasons for making the change. Our country is engaged in a broad technical assistance program to aid other nations in taking advantage of technology and raising their living standards. Our experts are finding considerable difficulty in dealing with systems foreign to their own.

Many other nations are having difficulty with our instruments because they are labeled in the archaic English system.

In the area of our foreign trade, we are beginning to be hurt by the fact that we do not produce and label our products in the metric system. Let me add to this point. We are engaged in a strong economic competition with the Soviet Union. It must be apparent to all that the uniformity of measurement systems between Russia and most of the world, including Western Europe, is an enormous advantage to the Soviets and a handicap to us. Further strengthening of the European Common Market will increase this problem. Our country also has adopted a system of military aid to our allies and friends. The effectiveness of this program has suffered, I have little doubt, from the incompatibility of measuring systems.

In brief, a dynamic country like ours where new commodities are adopted incessantly and where inventories are replaced periodically has the capability of executing a change in its measurement system. What we need is a procedure by means of which the change can be carried out most expeditiously with the least cost, the least confusion, and the least opposition. Such a change will eventually allow us to give better technical training in our schools because of the relative simplicity of the system.

For certain areas of our economy, the changeover would give no apparent advantage. For other areas, an abrupt changeover would prove very costly and time consuming. In land measurement, for example, it may take scores of years with the American title history as it is to convert it to a metric classification; and problems such as this may exist in other fields, but there is no need for instant and immediate conversion in these circumstances. In many areas, on the other hand, the change could be carried out with little or no trouble. I believe we ought to begin promptly to study the problem and to see whether a plan can be developed for an orderly and effective transition.

Accordingly, I propose to request the Director of the National Bureau of Standards to establish an advanced planning group to assemble all available documentation and to identify possible courses of action.

Through our advisory committee's contacts with business of all sorts, we will establish special industry committees to tell us of their special problems, and within the Department of Commerce the problems will be studied with a view toward determining, among other things, whether Government activities can be metricized and whether there are special legislative requirements which must be satisfied.

The work of the existing AAAS Committee on Metric Usage will receive the important consideration which it deserves, as well as that of the parallel committee of the British Association for the Advancement of Science. We will examine the experience of India and Japan in the fairly recent shift to the metric system in those countries. Japan's experience would be especially pertinent since that country faces a number of advanced technological problems similar to those faced by us.

If there should be a parallel committee in other countries using the English system, we would want to make our studies cooperative in order to insure continued effective cooperation and the avoidance of duplication.

In the meantime, the National Bureau of Standards will continue its extensive program of providing calibration services in both the English and the metric systems. The calibration system of the Bureau will be expanded and services in both systems will be available as long as any important segment of our society requires calibration in either system.

One recent improvement in the English measurement system was announced last winter by all of the national standards laboratories of the English-system-using nations. I refer to the agreement to define the inch as 25.4 millimeters, thus removing important minute discrepancies in the inch definitions of different countries. This agreement conforms to the recommendations of the American Standards Association and of the AAAS. This uniform and simplified definition of the international inch will provide for better measurement in communication between English-speaking nations, and because there is now an agreed mathematical relationship between the English system and the metric system, will make it easier to move from the English to the metric system.

In the meantime also I hope that a number of industries on their own will study the problem of converting to the metric system and advise us on their examination. Wherever possible I hope that they will take voluntary steps toward the effective transition to the metric system as has the pharmaceutical industry. I am fully aware of the leadtimes that may be required and the other problems that must be solved. But industry and science can contribute to the solution of these problems and I hope this will be done.

TWELFTH ANNUAL GASEOUS ELECTRONICS CONFERENCE

THE TWELFTH annual Gaseous Electronics Conference will be held at the National Bureau of Standards on October 14, 15, and 16, 1959. Sponsored jointly by the Bureau and the Division of Electron Physics of the American Physical Society, the conference will emphasize the physics of collisions in atomic systems. The keynote lecture on this subject will be given by D. R. Bates of Queen's University, Belfast, Ireland.

In addition to invited papers, already scheduled, short contributed papers of particular interest in the gaseous electronics field will be presented. Inquiries concerning the program and other conference arrangements should be directed to the conference secretary, L. M. Branscomb, Atomic Physics Section, National Bureau of Standards, Washington 25, D.C.

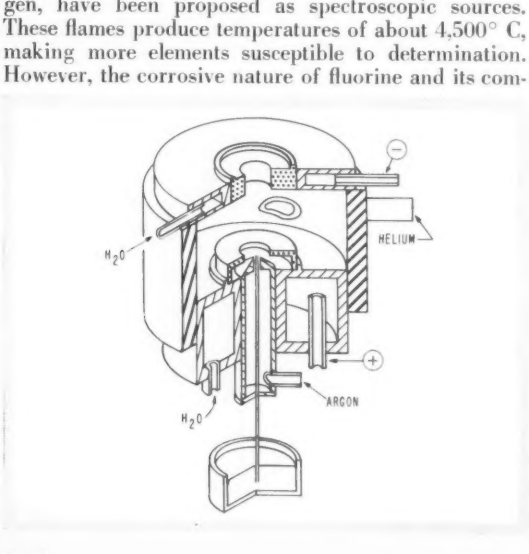
PLASMA JET AS A SPECTROSCOPIC SOURCE

Temperatures up to 8,000° C can be obtained with the plasma jet for spectral excitation of the elements vaporized from a solution. Standard solutions are used to compare the relative emission intensities in making quantitative determinations.

A MODIFIED plasma jet has been developed and is being investigated at the National Bureau of Standards as a spectroscopic source in work [1] sponsored by the Navy Bureau of Aeronautics. Producing temperatures up to 8,000° C in this application, the modified plasma jet offers promise as a means of excitation for the spectographic analysis of complex alloys.

The plasma-jet concept involves heating a gas stream to extremely high temperatures by constricting an electric arc passing between two electrodes. The hot ionized gas comprising the arc plasma emerges through an opening in the cathode as a flamelike jet, thereby giving the device its name. For spectrochemical analysis, a solution of the test specimen is drawn up a capillary tube and is sprayed into the arc plasma. The plasma jet vaporizes the solution and excites the elements to emit spectra that would not ordinarily be obtained at lower temperatures with chemical flames.

Flames have been favored for the spectrochemical analysis of solutions since the pioneering work of Kirchoff and Bunsen in 1860. Two of these sources which are generally used are the hydrogen-oxygen and acetylene-oxygen flames. Both flames provide a maximum temperature of about 3,000° C. However, this temperature is not sufficient to excite all of the elements in solution. Within the last few years two new flames, the hydrogen-fluorine and cyanogen-oxygen, have been proposed as spectroscopic sources. These flames produce temperatures of about 4,500° C, making more elements susceptible to determination. However, the corrosive nature of fluorine and its combustion product, hydrogen fluoride, present a serious problem in the handling of these gases. Cyanogen does not have these disadvantages and may prove useful as it becomes commercially available.



Simplified diagram of plasma jet. Shown are the inner chamber, cathode with its opening (top), and anode. Test solution (bottom) is drawn up through the capillary tube of the atomizer and sprayed into the arc plasma passing between electrodes. A hot flamelike jet emerges from the cathode opening.

Recently the plasma jet has been applied in several laboratories for high-temperature studies in physics and chemistry and as a source of high-temperature, high-velocity gas streams for wind tunnels. The plasma-jet units developed for these conditions have been operated at currents from 50 to many hundreds of amperes. As a result, jets several inches long and temperatures as high as 40,000° C have been produced. However, for application as a spectroscopic source, neither extremely high temperatures nor a very long flame are necessary.

In an effort to develop new spectrometric methods for analyzing complex alloys, M. Margoshes and B. F. Scribner of the NBS staff designed a plasma jet to be operated on conventional d-c power supplies. With a dc of 15 to 20 amp, the plasma jet reaches a maximum temperature of about 8,000° C, sufficient to excite all elements in solution. The spectra of the test samples are compared with those of standard solutions in making a quantitative determination. In preliminary tests, iron, chromium, and nickel, the major constituents of stainless steel, were determined with a 2-percent coefficient of variation.

The high temperature produced by the plasma jet is due to the thermal "pinch" effect. Heat from the d-c arc is concentrated into a narrow region between the electrodes by helium gas, acting as a "coolant".

The temperature of the plasma is highest at the cathode, decreasing as the hot gas expands and radiates energy. The maximum temperature is dependent on such factors as the diameter of the cathode opening, the current, and the coolant used and its flow rate.

In the plasma-jet device developed for spectroscopic use, an argon-operated atomizer is held within a coaxial water-cooled chamber and is protected by a graphite cap which serves as the anode. The atomizer and anode are enclosed within a hard rubber cylindrical case at one end. At the other end, a graphite disk with a central opening is held by a water-cooled brass ring. This disk serves as the cathode. Helium is introduced tangentially into the rubber case as a coolant for the d-c arc. Contained inside the atomizer is a 20-gage stainless-steel hypodermic needle through which the test solutions flow to the atomizer tip. The arc excites the spectral elements as the solution is sprayed from the atomizer and anode through the cathode opening.

Further studies are in progress to observe the characteristics of the modified plasma jet and to improve its accuracy and sensitivity as a spectroscopic source.

[1] *For further details, see* The plasma jet as a spectroscopic source, *by M. Margoshes and B. F. Scribner, Spectrochim. Acta* 138 (1959).

THERMAL DEGRADATION OF COLLAGEN

TO PROVIDE basic information on the properties of leather, the Bureau is investigating the processes that occur in the breaking down of collagen, the white connective tissue of hides from which leather is made.[1] In recent experiments conducted at high temperatures in both a dry and moist atmosphere, J. M. Cassel has developed extensive data on the processes of collagen deterioration. These results are expected to be of value in devising methods for making leathers more resistant to presently known degradation environments.

Collagen is a complex protein containing 17 different amino acids. The fibrous matrix and molecular structure of this natural polymer impart to leather many of its unique properties, such as tensile strength, elasticity, toughness, and pliability. The tendency of finished leathers to lose some of these properties and to deteriorate under certain environmental conditions is of prime concern to tanners, leather manufacturers, and industrial users of leather. The present investigation was therefore undertaken to detect the parts of the fundamental protein structure most susceptible to attack.

The material from which the various degradation products were derived was prepared from raw steerhide. The grain and flesh layers were split off and discarded. The corium, or inner layer, was ground to a powder and freed of extraneous fats, salts, and proteins by a series of aqueous and organic extractions.[2] The final product was then ground in a mill to pass a 4-mm sieve.

Procedures to degrade the collagen included heating in oxygen, air, and vacuum to temperatures as high as 170° C for a period of 3 days in a dry atmosphere; and in air at 100° C for 3 days at humidities varying from 20 to 90 percent. Collagens thus degraded were examined for: (1) Changes in moisture absorptive capacities and in amino acid content as detectable with paper chromatographic and colorimetric analyses;[3] and (2) formation of water-soluble fractions in what initially was a completely insoluble material. Since the water-soluble fractions were formed as a direct result of the degradation exposures, these fractions were examined for acidity, viscosity, ease of nitrogen precipitation, and aminonitrogen content. From these data, estimates of molecular size were made and types of chain scission were determined.

The exposures in a dry atmosphere reduced water absorptive capacity, produced water-soluble fractions of relatively low molecular weight, and caused considerable destruction of the amino acid residues—methionine, serine, threonine, and tyrosine. Greatest degradation was apparent in oxygen.

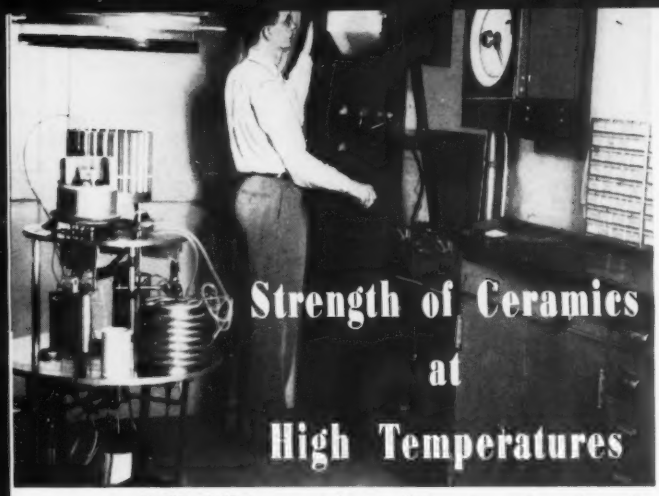
On the other hand, collagen heated at high humidities resulted in little amino acid destruction but relatively large amounts of water-soluble material. The molecular weight of this material was slightly larger than that formed as a result of heating in the absence of moisture. The chain scission which occurred in the presence of moisture was principally of a hydrolytic type, while that occurring in the absence of moisture and in the presence of oxygen was of an oxidative type.

The techniques followed in the present work will be employed to investigate the breakdown of hide substance in the presence of nonprotein ingredients such as tannins and greases. A study is now being made of the effects of gamma radiation on collagen. The data from this research will be integrated with the results of the present study to advance knowledge of the mechanisms involved in leather deterioration.

[1] *For further technical details, see* Degradation of collagen by heat, by James M. Cassel, *J. Am. Leather Chemists' Assoc.* **53**, *507 (1958)*.

[2] Studies on the purification of collagen, *by J. M. Cassel and J. R. Kanagy, J. Research NBS* **42**, *557 (1949)*; J. Am. Leather Chemists' Assoc. **44**, *424 (1949)*; *NBS Tech. News Bul.* **34**, *10 (1950)*.

[3] A paper chromatographic analysis for collagen and collagen derivatives, *by James M. Cassel, J. Am. Leather Chemists' Assoc.* **51**, *223 (1956)*; *NBS Tech. News Bul.* **40**, *65 (1956)*.



Strength of Ceramics at High Temperatures

Equipment for modulus of rupture measurements on aluminum oxide single crystals. Furnace (left center) is partially dismantled to show specimen, heater, and water-cooled shell (lower center). Bell jar (upper left) is used for vacuum conditions. H. S. Parker, engineer, reads record of furnace temperature. Temperature of the furnace is graphically recorded.

CERAMIC materials are being investigated at the Bureau for the Air Force to develop a better understanding of the factors controlling their strength at elevated temperatures. As part of this program, the strength of aluminum oxide single crystals (sapphire and ruby) has recently been studied [1] by J. B. Wachtman, Jr., and L. H. Maxwell at temperatures between 600° and 1,000° C. The study supports the theory that, in properly oriented crystals, plastic deformation relieves stress concentrations, thus increasing crystal strength.

Technological advances have brought about increased industrial use of ceramic materials such as aluminum oxide. Because of their heat- and corrosion-resistant properties, these materials find application in aerodynamics where high-temperature load-bearing structures are required. Ceramics are also of special interest in electronics as electrical insulators.

The widespread use of these ceramic materials makes it desirable to investigate the factors controlling their strength at elevated temperatures. However, a study of pure polycrystalline ceramics is complicated by grain boundaries and by interaction of the single crystal grains. To eliminate these complicating factors, the Bureau has been studying the properties of single crystals and their effects on polycrystalline materials.

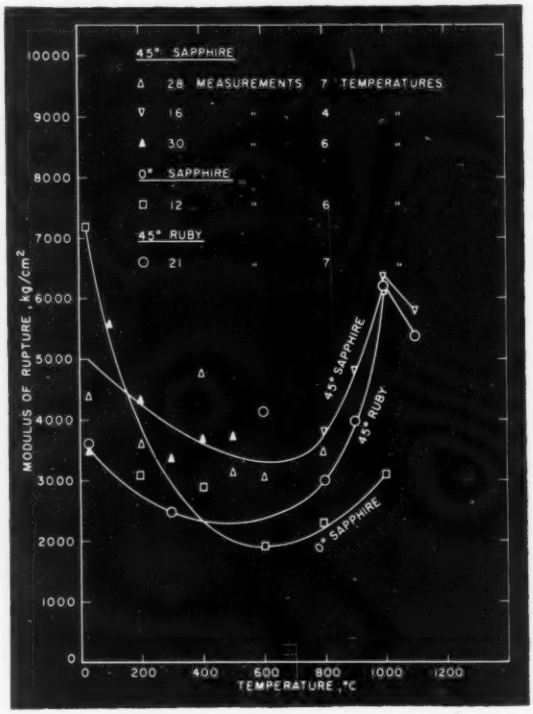
The strength of polycrystalline aluminum oxide is known to decrease with increasing temperature above 800° C. However, in one set of measurements on single-crystal aluminum oxide, crystal strength increased with temperature in this range.[2] It was suggested that this might be caused by very small amounts of plastic deformation. That is, failure may have begun at points where the local stress was much greater than the average applied stress. Plastic deformation might then have reduced the degree of stress concentration with the result that a larger average stress could be sustained before failure occurred.

To test this hypothesis, both sapphire and ruby crystals were favorably oriented (45°) for plastic deformation, and modulus of rupture was measured on the crystals as a function of temperature up to 1,000° C. For comparison, like studies were made on unfavorably oriented (0°) sapphire.

The apparatus used for the modulus-of-rupture measurements was recently developed [3] at the Bureau in similar studies on uranium and thorium oxides. It permits measurements to be made in a vacuum, or in an inert or oxidizing atmosphere at temperatures up to 1,200° C with a controlled loading rate.

In this apparatus, the bar specimen is supported at its ends and loaded near its center within a resistance-type furnace. Force is applied by a ceramic yoke which extends through the floor of the furnace to a lever. An automatic-loading system periodically adds a predetermined amount of lead shot to a bucket on the lever. When the specimen breaks, a trip switch is thrown which prevents further shot from being added.

The resulting data are consistent with the hypothesis that microscopic plastic deformation at stress concentrations is responsible for the rise in strength, which actually begins above 600° C for the single crystals. Observations which support this assertion are (1) the large increase in strength for both 45° sapphire and 45° ruby and (2) the large scatter in strength values obtained at temperatures below 600° C as compared



Strength-versus-temperature curves for aluminum oxide single crystals.

with those at higher temperatures. It seems reasonable that partial relief of stress concentrations should decrease the scatter in strength values.

The decrease in strength of polycrystalline aluminum oxide above 800° C, which is in contrast to results for the single crystal, was thought to result from stress concentrations caused by grain boundary slip. Support for this idea was obtained by measuring Young's modulus and the internal friction as a function of temperature for the polycrystalline material. Both measurements indicated stress relaxation characteristic of grain boundary slip. This relaxation of shear stress on grain boundaries should cause increase in local tensile stress at other points. Consequently the specimen would not be able to sustain as large a value of average applied stress.

Although the theory of plastic deformation is complex, research scientists are making progress in studying the effects of this mechanism in metals and metal oxides. An explanation based on "dislocations", a type of imperfection in a crystalline solid, offers a clearer understanding of the process.

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[1] *For further technical details, see* Strength of single crystal sapphire and ruby as a function of temperature, *by J. B. Wachtman, Jr., and L. H. Maxwell, J. Am. Ceram. Soc. (in press).*

[2] On the strength of polycrystalline and single crystal corundum, *by E. A. Jackman and J. P. Roberts, Trans. British Ceram. Soc.* **54,** *389 (1955).*

[3] Effect of particle size on bulk density and strength properties of uranium dioxide specimens, *by M. D. Burdick and H. S. Parker, J. Am. Ceram. Soc.* **39,** *181 (1956).*

BUREAU PHYSICISTS WIN GUGGENHEIM AWARDS

Dr. Chan-Mou Tchen

DR. CHAN-MOU TCHEN of the Mathmetical Physics Section and DR. MICHAEL DANOS of the High Energy Radiation Section have been chosen to receive Guggenheim Memorial Foundation Awards. The fellowships are designed to aid persons of proven research ability in furthering their studies in their chosen fields.

Dr. Tchen will study plasma physics and magnetohydrodynamics, principally at the Max Planck Institute for Physics and Astrophysics, Munich, Germany. Dr. Danos will investigate the quantum mechanical foundation of the phenomenological theory of the photonuclear giant resonance at the University of Heidelberg.

Dr. Tchen was born in Fenghua, China. He received his Doctor of Technical Sciences degree (cum laude) from the Technical University of Delft, Netherlands, in 1947.

In 1947, Dr. Tchen came to the Bureau as a research physicist. He is author of numerous research papers on such subjects as turbulence, instability, heat transfer, viscoelasticity, macromolecules, stochastic processes, diffusion, statistical mechanics, and plasma physics. Dr. Tchen is a member of the

Dr. Michael Danos

American Physical Society, the Washington Academy of Sciences, Sigma Xi, Sigma Pi Sigma, and the American Geophysical Union.

Dr. Danos, who was born in Riga, Latvia, in 1922, attended the University of Riga, majoring in electrical engineering, and received his M.S. from the Technical University of Hanover, Germany, in 1948, and his Ph. D. in physics from the University of Heidelberg, Germany in 1950.

Before coming to the Bureau in 1954, Dr. Danos was an assistant physics instructor at the University of Heidelberg and a research associate for the Columbia University Radiation Laboratory.

At the Bureau, Dr. Danos has been engaged in theoretical research in nuclear and radiation physics and in the development of a microwave electron spin magnetometer for time-dependent inhomogeneous magnetic fields.

Dr. Danos has published numerous research papers, and is a member of the American Physical Society, the American Association for the Advancement of Science, and the Federation of American Scientists.

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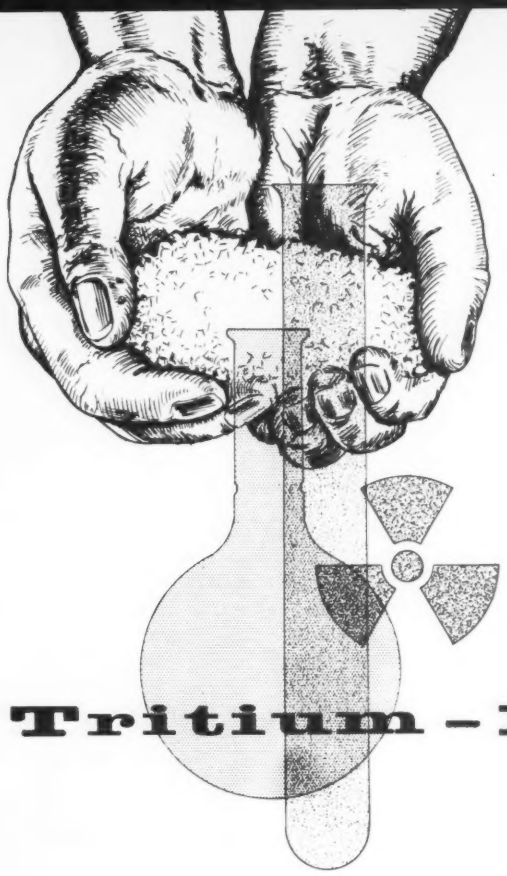


Tritium-Labeled Sugars

SUGARS and sugar derivatives having tritium—the radioactive isotope of hydrogen—substituted for hydrogen in a specific site within the molecule are being prepared by the Bureau in a project sponsored by the Division of Research of the Atomic Energy Commission. At present, all of the common sugars labeled with tritium at carbon 1 are available for distribution to other laboratories.[1] These sugars, identical with ordinary sugars except for the radioactive tritium atom, should prove to be a valuable tool in tracer research on chemical, medical, and biological problems.

Methods of analysis and multipurpose equipment which facilitate chemical application of tritium, as well as practical procedures for the synthesis of tritiated carbohydrates, have been designed. These developments are part of a broad research program on radioactive carbohydrates[2] now being conducted by H. S. Isbell and associates of the Bureau's organic chemistry laboratories. One phase of this program, initiated in 1950, has made possible the synthesis and distribution of a wide variety of carbon-14-labeled carbohydrates[3] to research workers in other laboratories.

Tritium is one of the products from nuclear reactors. It has a combination of properties that makes it an extremely versatile research tool. Although it is highly radioactive, its low-energy beta radiation is so weak that it can be blocked by a sheet of paper. Thus, when tritium is incorporated in nonvolatile materials, it presents little or no radiation hazard, and is relatively safe to use. One of the cheapest radioisotopes (only one eleven-thousandth as expensive as carbon-14), tritium can be detected in extremely small quantities by means of its radiation. As little as one-billionth of a milligram of tritium oxide (tritiated water) can be readily measured.

Because tritium is the radioactive isotope of hydrogen—a constituent of nearly all organic compounds—and enters into the same chemical reactions as hydrogen, its possible uses and applications in organic and biological chemistry are practically unlimited.[4] In the form of tritiated water or other tritiated reagents, tritium can react with a wide variety of organic compounds. Thus, a few tritiated starting materials enable the chemist to prepare numerous radioactive compounds for use in chemical and biological studies. Once incorporated in a stable molecule in place of hydrogen, the isotope serves as a 'tag' that can be used to trace the course of an atom or a molecule through an entire series of complex chemical and physical processes.

Despite these advantages, tritium's widespread use has been hampered by lack of convenient methods of analysis. If a radioisotope is to have maximum usefulness in chemical and biological studies, it must be possible to measure its radioactivity rapidly and simply. Analytical methods for tritium, such as the counting of gaseous samples in ionization chambers or dissolved samples in liquid scintillation counters, are time consuming and require elaborate equipment.

Analytical Method

The Bureau has now developed a practicable method[5] for the routine analysis of tritium in water-soluble, nonvolatile materials. In this method, the radiation from a tritium compound, which is incorporated in a film, is measured in a "proportional counter". Because of its simplicity, the method should make possible numerous procedures employing tritium as an analytical reagent.

In the first step of the analysis, the radioactive substance is combined with a water solution of a thickening agent (preferably carboxymethylcellulose). This radioactive mixture, on a counting plate, is then converted to a film by evaporating the water. The radiation from the film is measured in the windowless, gas-flow, proportional counter, which is available in most laboratories where radioactive materials are used. The amount of radioactivity is easily calculated from the net count, the weight of the film, and an empirical constant determined with a substance of known radioactivity.

The radioactive film, though very thin, is "infinitely thick" to the weak radiation from tritium. That is, the radiation from the lower part of the film is completely absorbed, and a constant proportion from the upper part emerges into the sensitive counting area of the proportional counter.

The presence of the thickening agent has two effects: it promotes formation of a uniform film, and it prevents the radioactive substance from crystallizing on the counting plate—a process that can lead to errors in measurement, or even to radioactive contamination of the counter.

Closed-System Apparatus

Because tritium is a gas and must therefore be used in closed systems, the development of a general-purpose, closed-system apparatus for conducting a variety of operations [6] was one of the first considerations in the tritium program. To this end, improved procedures and special techniques were worked out for such operations as filtration and crystallization within a vacuum system. The essential feature of the resulting apparatus is a vacuum manifold—a central glass vacuum line—to which numerous pieces of specialized apparatus can be attached as needed. By interchanging the connections, various operations can be conducted in the one composite apparatus.

Because tritium in high concentration decomposes organic compounds by the energy of its radiation, it is frequently diluted with hydrogen before use. The apparatus provides a convenient method for making such dilutions, as well as for removing samples of gas for radioactivity determinations, and for circulating tritiated hydrogen in such procedures as catalytic reductions. The gas is circulated by a gas-transfer pump containing mercury (Toepler pump).

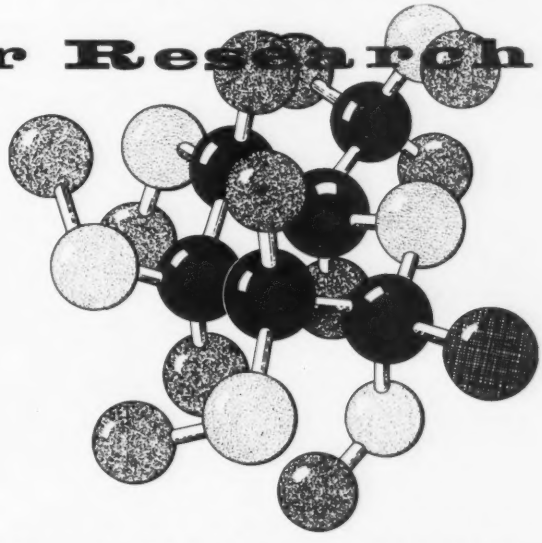
Two important tritium reagents, tritiated water (tritium oxide) and tritiated lithium borohydride, can be synthesized in this apparatus. As both of these compounds have readily available tritium, they are used to introduce tritium into organic compounds, and to synthesize tritiated materials. To prepare tritiated water, tritium gas is passed from a gas burette through a copper oxide furnace heated at 500° C. The tritiated water that forms is collected in a water trap cooled in a freezing mixture. Finally, the trap containing the tritiated water is isolated, and the water is stored until needed.

Tritiated water can be used to study such practical problems as humidity control, adsorption of moisture in materials of all types, permeability of films to moisture, formation of hydrates, and a variety of other chemical reactions that involve water.

Tritiated lithium borohydride, another versatile tritium reagent, is prepared from lithium borohydride and tritium gas. First lithium borohydride is converted to a fluffy, porous form suitable for tritium exchange. To accomplish this, commercial lithium borohydride is dissolved in a solvent, the solution is filtered, and the solvent is removed by "freeze-drying" (low-temperature vacuum drying). This entire operation is conducted in the closed system because lithium borohydride reacts rapidly with the moisture of the air. The finely divided powder is then heated with tritium gas at 200° C. At this temperature, tritium-hydrogen exchange occurs, yielding the tritiated lithium borohydride. The excess tritium-hydrogen mixture may be stored in a flask for future use, or converted to tritiated water in the copper-oxide furnace. Numerous other reactions and processes, including syntheses of tritium-labeled carbohydrates, can be conveniently carried out with the same apparatus.

Syntheses

One of the objects of the program on chemical applications of tritium is to develop methods for the synthesis of carbohydrates position-labeled with tritium.



Structural model of glucose molecule labeled with tritium at the carbon 1 site (right diagonally shaded ball). See configurational formula on page 133.

In general, the synthesis of each substance presents problems of its own, and separate syntheses must be developed for each different position-labeled version of the same compound.

Glucose having tritium attached to carbon 1 is prepared by the reaction of gluconic lactone with tritium-labeled borohydride in the general-purpose apparatus. The reaction can be carried out conveniently by using a hypodermic syringe to inject the sugar, through a rubber stopper, into a flask containing lithium borohydride-*t* in dry tetrahydrofuran. When the reaction

is completed, excess borohydride is decomposed by injecting a small amount of dilute acetic acid into the flask, and the excess tritium-hydrogen is removed by the Toepler pump. The flask is then removed from the apparatus, and the tritium-labeled glucose is purified by passing the glucose solution through an ion-exchange column and repeatedly concentrating a methanol solution of the glucose to dryness. After this treatment, the glucose-1-*t* is further purified by recrystallization.

As glucose is biologically the most important of the simple sugars, the Bureau is now developing methods for synthesizing glucose labeled with tritium at each of the six carbon atoms. Other sugars and sugar derivatives will be prepared as synthetic methods are developed. Although several methods of synthesis are possible for each labeled sugar, the Bureau is seeking methods that produce the highest yields in the shortest time.

In addition to position-labeled simple sugars, other potentially useful tritium-labeled materials, such as tritiated inulin, are being made. Used clinically for many years to study kidney function, inulin is injected into the bloodstream, and its elimination in the urine is measured. Determination of inulin by chemical analysis is laborious but can be avoided by using C^{14}-labeled inulin (a substance previously prepared by the Bureau)[7] and determining its elimination by means of radioactivity measurements of the urine. A new tritiated form of inulin, considerably cheaper than the corresponding C^{14}-labeled product, may prove to be an even better material for the study of kidney function.

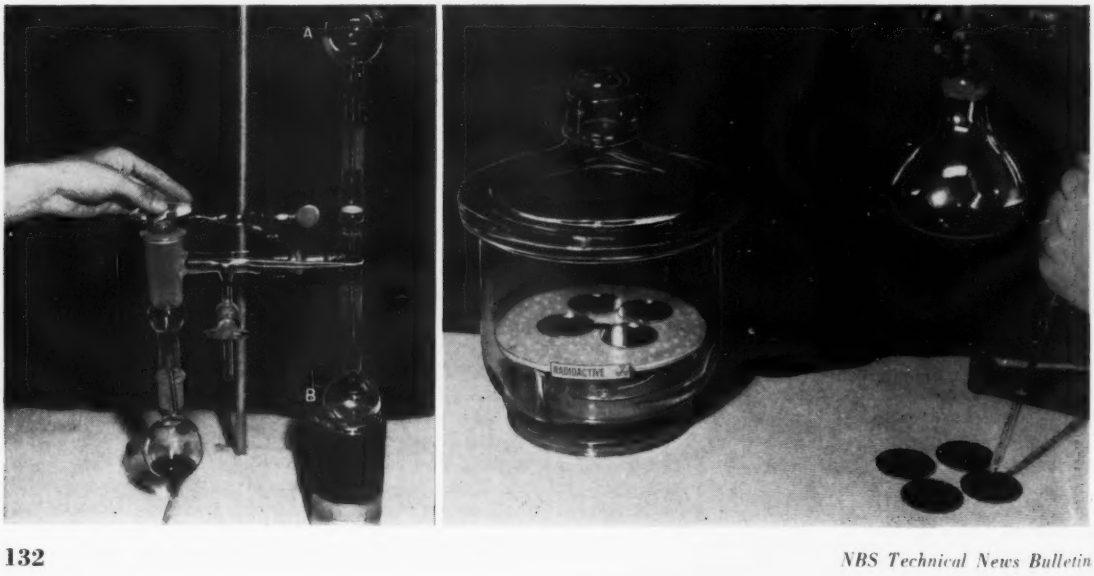
Study of Isotope Effects

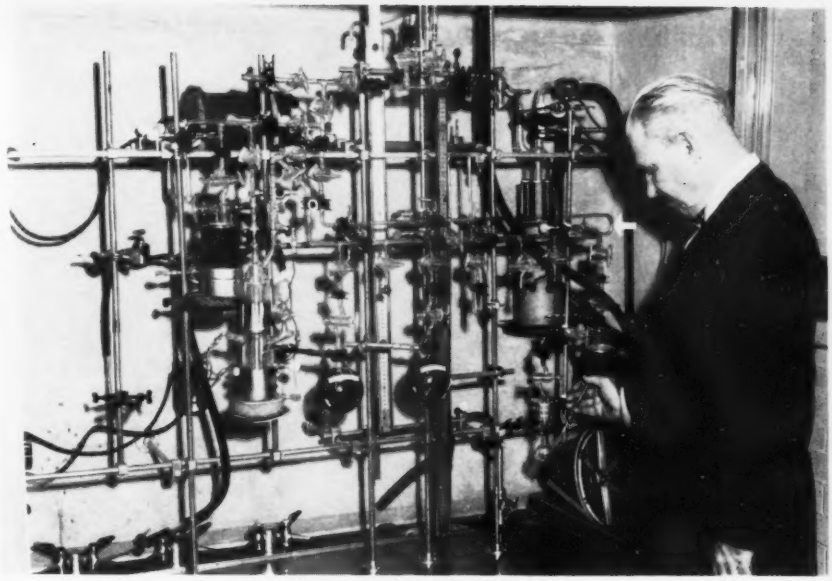
One advantage of using an isotope in research is that its physical and chemical behavior is very similar to that of the common form of the element. However, an isotope is not identical in behavior to the common form. For example, there are slight differences in rates of reaction between ordinary carbon and carbon-14, and larger differences in rates between ordinary hydrogen and tritium because tritium is three times as heavy as hydrogen. The rate difference is detectable chiefly when a chemical reaction takes place at the site of an isotope bond. If the isotope is located elsewhere in the molecule, it has little effect on the rate of reaction.

Unless this difference in behavior can be predicted, it may limit certain uses of isotopes. For example, in locating the position of a radioactive atom within a molecule, the compound is broken into several simpler ones by using oxidizing agents. By the specific activity of the fragments produced, the location of the radioactive atom can be detected. If the radioactive molecule should degrade at a different rate than the nonradioactive form, the results and conclusions drawn from the degradation study would be misleading.

Although an isotope effect is disadvantageous for some purposes, it may furnish data on the course of a reaction by indicating whether an isotope bond is involved. Employing tritium-labeled materials, the Bureau is studying the mechanism of such reactions as the reduction of lactones, and the oxidation of sugars with bromine, chlorous acid, and oxygen. Use of tritiated compounds in these and other reactions should supply detailed information on reaction mechanisms.

Left: **Apparatus designed by the Bureau for filtering and recrystallizing tritiated compounds in a small closed system. At start of purification process, flask C contains a solvent, flask A (initially in the lower position) contains the solid to be recrystallized. Solvent is distilled from C to A and the solid is dissolved. Then A and B are rotated through 180 deg, flask B is cooled (as shown) and the solution is filtered through a fritted glass disk into B.** ***Right:*** **For quantitative determination of tritium content, sample in a carboxymethylcellulose solution is converted to a film under infrared light (right). After this radioactive film has been conditioned at controlled humidity (left), it is assayed in a windowless proportional counter.**





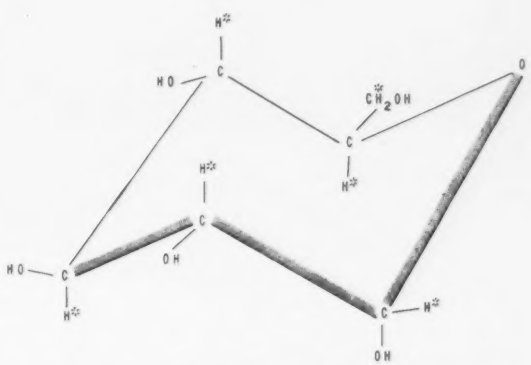
General-purpose manifold for handling tritium in a closed system. By attaching and interchanging specialized equipment to the manifold (indicated by arrow), many different operations can be conducted. Here H. S. Isbell introduces a reagent to begin a synthesis.

A reaction uniquely suited to the study of the isotope effect is being carried out with various carbon-14- or tritium-labeled forms of a six-carbon carbohydrate derivative, D-mannitol. When this compound is oxidized by the microorganism, *Acetobacter suboxydans*, it yields the sugar, D-fructose. D-Mannitol is a substance in which the two halves of the molecule are alike, so that the enzymes created by *A. suboxydans* have a choice of two suitable sites for oxidation: carbon 2 or carbon 5. In a position-labeled D-mannitol, one-half of the molecule contains a radioactive isotope. If its presence does not influence the rate of oxidation, then the resulting D-fructose must have radioactivity equally divided between the two halves. The results of the study show [8] that there is no isotope effect in the oxidation of D-mannitol-1-C^{14}, but that there is a small effect (about 3%) in the oxidation of D-mannitol-2-C^{14}. Studies of an isotope effect in the oxidation of D-mannitol-3-C^{14} and of the various tritium-labeled D-mannitols are in progress. The results will aid in the interpretation of the mechanism of enzymic oxidation by *A. suboxydans*.

Tritiated Reagents

Part of the program on chemical applications of tritium will be devoted to the synthesis and use of tritiated reagents. It is believed that these compounds offer a vast opportunity for research on analytical methods, structural studies, and reaction mechanisms. In connection with the carbon-14 program, a procedure has been developed for conducting methylations with C^{14}-labeled methyl iodide.[9] Tritiated methyl iodide will be similarly employed. Reduction with tritiated lithium borohydride, following oxidation procedures, provides an excellent method for introducing a radioactive label at a specific point in a molecule. Wherever a carbonyl group (carbon doubly bonded to oxygen) can be formed in a molecule, tritium can be introduced by reaction with tritiated lithium borohydride. This technique provides a means for locating the oxidation sites, i.e., the critical points at which oxidation has taken place in a molecule. Studies utilizing the technique will aid in solving such technological problems as the determination of the chemical changes in cellulose fibers caused by bleaching agents, air, light, or aging.

The Bureau has in progress numerous other research projects on tritium and its applications. The direction



Configurational formula for D-glucose. Syntheses are being developed in which tritium will be substituted for hydrogen at each of the sites marked by an asterisk.

of the program will be determined, to some extent, by the needs of other research workers. As in the program on carbon-14-labeled carbohydrates, it is believed that the tritium work will greatly facilitate the work of other laboratories and contribute fundamentally to the application of radioactive isotopes in organic and analytical chemistry.

Tritiated Sugars and Sugar Derivatives Now Available at NBS

D-glucose-1-*t*	L-arabinose-1-*t*
D-glucose-6-*t*	D-ribose-1-*t*
D-glucitol-1-*t*	D-xylose-1-*t*
D-glucitol-6-*t*	D-lyxose-1-*t*
D-galactose-1-*t*	lactose-1-*t*
D-galactitol-1-*t*	maltose-1-*t*
D-mannose-1-*t*	L-rhamnose-1-*t*
D-mannitol-1-*t*	D-*glycero*-D-*gulo*-aldoheptose-1-*t*
D-arabinose-1-*t*	inulin-*t*

[1] *Tritiated sugars may be obtained by purchase order to the National Bureau of Standards, Washington 25, D.C. They are priced at $10 per 100 microcuries.*

[2] *A complete list of the Bureau's publications and patents on this project will be furnished on request to the Organic Chemistry Section, National Bureau of Standards, Washington 25, D.C.*

[3] Position-labeled radioactive sugars, *NBS Tech. News Bul.* **36,** *56 (1952).*

[4] Use of carbon-14 and tritium for the study and characterization of cellulose and other polysaccharides, *H. S. Isbell, H. L. Frush, and J. D. Moyer, Tappi* **40,** *739 (1957); AEC Report No. NBS-4967.*

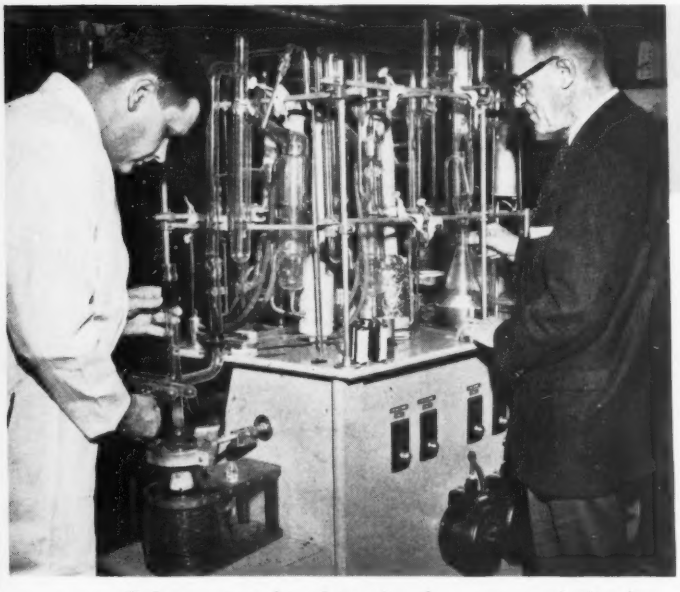
[5] Tritium-labeled compounds. I. Radioassay of tritium-labeled compounds in "infinitely thick" films with a windowless, gas-flow, proportional counter, *H. S. Isbell, H. L. Frush, and R. Peterson, J. Research NBS (in press).*

[6] Tritium-labeled compounds. II. General-purpose apparatus, and procedures for the preparation, analysis, and use of tritium oxide and tritium-labeled lithium borohydride, *H. S. Isbell and J. D. Moyer, J. Research NBS (in press.)*

[7] Process for labeling polysaccharides with C^{14} and products resulting therefrom, *H. S. Isbell, U.S. Patent No. 2,653,931, Sept. 29, 1953;* Carbon-14 carboxy-labeled polysaccharides, *J. D. Moyer and H. S. Isbell, J. Research NBS* **61,** *71 (1958) RP2886.*

[8] Isotope effect in oxidation of D-mannitol-2-C^{14} *by Acetobacter suboxydans, H. L. Frush and L. J. Tregoning, Science* **128,** *597 (1958).*

[9] Micromethylation of polysaccharides, *H. S. Isbell, H. L. Frush, B. H. Bruckner, G N. Kowkabany, and G. Wampler, Anal. Chem.* **29,** *1923 (1957).*



Eqiupment used to determine the oxygen and nitrogen content of metals. J. T. Sterling adjusts vertical tube above the furnace (lower left) while L. L. Wyman takes readings on pressure gage.

STANDARD SAMPLES for GASES in METALS

EIGHT BARS of ferrous materials, whose oxygen and nitrogen content have been precisely determined, comprise a new type of standard sample now available from the Bureau. These reference materials, approximately 3 in. long and 1 in. in diam, are necessary for the calibration of chemical analytical equipment used to measure the gas content of various kinds of commercial steels. Low gas content is a characteristic of metals with desirable properties, and the acceptable amount of gases present in a metal is generally stipulated in procurement specifications for metals and metal products.

J. T. Sterling and L. L. Wyman of the chemical metallurgy laboratory employed the vacuum-fusion method [1] to analyze the standard samples that are now available. In this method a small furnace, containing a graphite crucible, is connected to a vacuum system by means of a vertical tube. When the furnace has been heated to a high temperature—usually about 1,600° C for steels—the metal sample, which had previously been placed inside horizontal side arms connected to the vertical tube, is maneuvered with a magnet until it falls into the hot crucible. As the sample melts, the gases liberated by the reaction of the graphite in the crucible are collected for analysis. The oxygen thus obtained is evaluated as carbon monoxide, and the nitrogen as molecular nitrogen.

In making the present standards available, the Bureau has inaugurated a program for providing gases-in-metals standards for a number of metallurgical products. Work is now in progress to develop standard samples for gases in titanium. The demand for such standards has increased in the last few years

TABLE 1. *Standard samples of iron and plain carbon steels for oxygen and nitrogen*

Sample number	Type	Additions			Composition, weight percent								
		Furnace	Ladle	Mold	O	N	C	Mn	P	S	Si	Cr	V
1040	Low-carbon, rimming	None	FeMn	None	0.018	0.003	0.03	0.3	0.01	0.04	0.002	0.004	<0.001
1041	Medium-carbon, high-manganese, silicon-killed.	FeMn	Hot metal, FeMn, FeSi.	None	.017	.004	.4	1.2	.02	.03	.3	.02	<.001
1042	Bessemer screw stock, rimming.	None	FeMn, S	FeSi	.017	.014	.1	0.7	.1	.2	.02	.006	.004
1043	Special, low-carbon, aluminum-killed.	SiMn, FeMn.	FeSi, Al	None	.002	.005	.2	.6	.01	.03	.09	.01	.001
1044	Low-carbon, silicon-killed.	None	FeMn, FeSi.	None	.009	.004	.2	.4	.02	.04	.1	.02	.001
1045	Medium-carbon, silicon-killed.	Spiegel	FeMn, FeSi.	None	.007	.004	.4	.5	.01	.03	.2	.01	<.001
1046	Open-hearth iron, rimming.	None	Al	None	.106	.005	.02	.02	.01	.02	.001	.01	<.001
1047	Similar to No. 1043 but higher in oxygen.	None	Al	Al	.017	.004	.2	.4	.01	.03	.03	.01	<.001

because improved specifications are required for the metals used in space vehicles.

The new reference materials are certified only for the elements, oxygen and nitrogen. However, the percentages of the other elements are given as a guide to the type of iron or steel. The standards are available from the Standard Sample Clerk, National Bureau of Standards, Washington 25, D.C.[2] The fee is $10 per sample. A certificate of analyses accompanying the materials lists the laboratories which have cooperated in this effort, and the various methods that have been utilized to determine the gas content in metals.

[1] Cooperative study of methods for the determination of oxygen in steel, *by John G. Thompson, Herbert C. Vacher, and Harry A. Bright. J. Research NBS* **18**, *259 (1937) RP976; Trans. AIME, Iron and Steel Division* **125**, *216 (1937)*.

[2] *Over 600 standard samples of chemicals, ores, ceramics, metals, and other substances are available from the National Bureau of Standards. Information is contained in Standard Materials, NBS Circ. 552 (third edition), which may be obtained by writing to the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. (35 cents).*

DICKINSON RECEIVES WILLIAMS AWARD

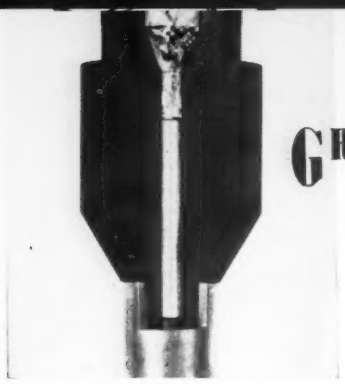
JOHN A. DICKINSON, Chief of the Codes and Safety Standards Section of the Building Technology Division, has been named winner of the Arthur Williams Memorial Award, presented annually by the American Museum of Safety to an individual or organization for "most outstanding achievement . . . in the interest of greater and improved industrial and general safety".

Mr. Dickinson was cited for "inspiring leadership in the formulation of American Safety Standards". The citation reviewed his long career in eliminating safety hazards while inspecting industrial establishments for an insurance company, preventing accidents in explosives manufacture with a chemical concern, and developing national safety standards and codes in his present position with the National Bureau of Standards. In cooperation with the American Standards Association, Mr. Dickinson has made major contributions to the safety codes for elevators and escalators, manlifts, logging and sawmills, power presses, electric lines, and lightning protection.

Born in Newark, N.J., in 1899, Mr. Dickinson studied mechanical engineering at Stevens Institute of Technology, and received an honorary degree in 1957. He joined the Bureau in 1919 as a safety engineer, and was made Assistant Chief of the Codes and Specifications Division in 1941. When this Division became the Codes and Safety Standards Section in 1947, Mr. Dickinson continued as Assistant Chief, and was named to his present position in 1955.

Mr. Dickinson is a member of the American Ordnance Society, the National Fire Protection Association, the Franklin Institute, the American Association for the Advancement of Science, and is charter member and executive committeeman of the American Society of Safety Engineers.

Articles on safety by Mr. Dickinson have appeared in such publications as *National Safety News, Safety Engineering, Factory, American Machinist, Safety News, Elevator World*, and *Standards*.



GROWTH of METAL SINGLE CRYSTALS

Left: **Closeup of seed (center) held firmly against main crystal (top) by graphite rod, a small portion of which is visible between the cut portions of copper pipe at bottom.**

IN ONE PHASE of basic corrosion research at the Bureau, metal single crystals are used to study the complex destructive processes of corrosion. These studies require specimens with a definite atomic arrangement, either in their surfaces or in certain crystallographic directions within the specimen. However, difficulty has been experienced in producing specimens having a preselected preferred orientation in combination with the desired cross-sectional shape.

A preferred crystallographic orientation can be obtained by growing crystals in a horizontal furnace; but specimens with a particular shape—cylindrical, for instance—can be produced only in a vertical furnace. By adapting the technique employed in the horizontal furnace for use in a vertical furnace, T. H. Orem of the corrosion laboratory has devised a means for growing crystals of the desired orientation in practically any shape.[1]

This method employs a procedure known as "seeding", which has been used to grow preferentially oriented crystals in a horizontal furnace. In this procedure, the seed—a small single crystal of the same metal as the main crystal—is attached to the main crystal in a boat-type container. The crystallographic orientation of the seed, which has been ascertained by X-ray diffraction methods, is fixed parallel to the orientation desired in the main crystal. This orientation is also parallel to the direction in which the metal is moved from a molten zone to a cool zone in the horizontal furnace.

Only a portion of the seed crystal is melted as the molten metal passes laterally through the horizontal furnace. The rate of movement is controlled to permit the molten portion of the seed to freeze in accordance with the orientation of the unmelted portion. Subsequently, as the molten mass slowly cools, the main crystal freezes in the same orientation. Efforts to produce this preferred orientation in a vertical furnace, however, have not been generally successful in the past because of the difficulty inherent in attaching and partially melting the seed in an upright position.

To overcome this difficulty, special equipment was designed. It consists of two cylindrical crucibles of graphite machined so that they can be joined together to hold the main crystal and the seed, respectively. A cooling device (a copper pipe about 2½ in. long with a wall thickness of ⅛ in.) is fastened to the lower portion of the seed crucible to conduct heat away from the bottom of the seed and thus prevent this portion from melting. A graphite rod, inserted in a cavity in the lower portion of the seed crucible, holds the seed in position against the bottom of the main crystal.

The assembly is lowered through the top of a vertical furnace to a position where only the upper part of the seed is melted. This position is ascertained with a thermocouple placed in the immediate vicinity of the assembly's cooling device. When the correct position has been established in a furnace of known heat content, the same percentage of seed can be melted in successive operations. It has been found that seeds melted in this manner can be used over and over again. It has also been found that the most desirable specimens can be produced when the crucible assembly is lowered in the furnace from the molten zone to the cool zone at a speed of one-half inch per hour. With this procedure, the solidifying metal not only assumes the orientation of the original seed crystal, but also freezes in any desired shape.

[1] *For further technical details, see* Growth of preferentially oriented aluminum single crystals, *by Theodore H. Orem, NBS J. Research* **60**, *547 (1958) RP2868.*

T. H. Orem places crucible assembly containing both seed and main crystal in vertical furnace used to grow single crystals with preferred orientation.

SEPARATION AND DETERMINATION OF PHOSPHATE, SILICATE, AND ARSENATE

A RAPID, accurate method for separating and determining small amounts of phosphate, silicate, and arsenate when they occur together in solution has recently been developed by W. S. Clabaugh and Audrey Jackson.[1] On the addition of ammonium molybdate, the three ions form heteropoly molybdate complexes, which, at high acid concentrations, can be extracted selectively for quantitative determination. The method should be useful in controlling the quality of materials such as chemicals, metals, glasses, and water that contain these salts as impurities.

Extraction, performed here by Audrey Jackson, is an important step in determining phosphate, silicate, and arsenate together in solution. The analysis depends on separating the complexes which the three ions form with ammonium molybdate.

This procedure has resulted from efforts to improve the test for phosphate in ammonium molybdate.[2] In attempting to limit interference from silicate and arsenate, the investigators found that all three ions can be determined in the same analysis, with a consequent saving in time and effort.

The sample to be analyzed for the three ions is first dissolved in water and a 10-percent solution of ammonium molybdate is added. Bromine water is then used to oxidize the arsenic to the quinquevalent state—tervalent arsenic does not form a heteropoly complex with molybdate. The acidity of the solution is adjusted to a *p*H of about 1.8 for the rapid formation of the complexes.

At this step in the separation process, the acidity is further increased. Ethyl ether is added and the mixture separated into an ether layer containing the phospho-molybdate complex and an aqueous layer containing the silicate and arsenate complexes. The ether layer is then washed with diluted hydrochloric acid and diluted with ether. Stannous chloride is added to produce the well-known molybdenum blue. The optical density of the solution, measured with a spectrophotometer, is referred to a calibration curve to determine the amount of phosphate present in the sample.

To separate the silico-molybdate complex from the remaining aqueous solution, the acid concentration is again increased and butanol is added. The silico-molybdate complex is extracted into the butanol layer while the arsenate complex remains in the aqueous layer. The butanol layer is then treated in the same manner as the ether layer in the first separation. Once the optical density of the solution has been measured, the amount of silicate present is determined from a calibration curve.

The arseno-molybdate complex is separated from the remaining aqueous solution by first adjusting the acid concentration to that used for the phosphate separation and then adding methyl isobutyl ketone. The amount of arsenate present is then determined spectrophotometrically.

The reliability of this technique was investigated by applying it to synthetic solutions containing known amounts of the three salts. Good agreement was obtained between the amounts added and the amounts determined.

Further investigation showed that accurate determinations of the three ions can be made when either chloride, nitrate, sulfate, or perchlorate is present in solution at concentration up to 5 g/100 ml.

[1] *For further technical details, see* Separation and determination of phosphate, silicate, and arsenate, *by W. S. Clabaugh and Audrey Jackson, J. Research NBS* **62,** *5 (1959) RP2934.*

[2] *Reagent Chemicals, American Chemical Society, Specifications 1955.*

HIGH PRESSURE EFFECTS ON SOLIDS

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Diamond holders used in high-pressure infrared absorption studies. In use, flat sides of these holders face one another, and very small diamonds are held at the centers. Area of each of the diamond faces is approximately 0.00015 in.^{2}. When 70 lb force is applied in this area, pressures between the diamond faces exceed 400,000 lb/in.^{2}.

SPECTROSCOPIC EVIDENCE has been found that changes in molecular structures occur under pressures of 30,000 atm.[1] The studies, by C. E. Weir and others, have brought out new information on the behavior of solids under pressure.

The basic characteristics of matter are determined by the nature of atomic and molecular interactions. Although the broader aspects of interactions between adjacent molecules are understood, a detailed evaluation has not been made of the disturbances produced by interactions between more widely separated molecules. The Bureau is studying these interactions as part of its program of basic research aimed at determining physical constants and properties of materials.

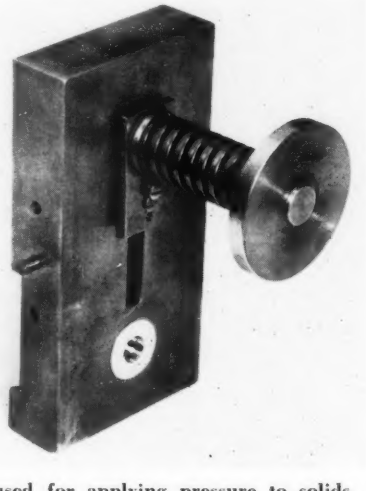
Infrared spectroscopy is used in this work because the frequencies of absorption bands are determined by the characteristics of the molecular units present, such as the masses of the oscillating units, the forces between them, and the force fields of adjacent molecules. Intensities of these bands are functions of the electrical properties (dipole moments) and the number of the individual oscillators, as well as their distance from one another. When a specimen is compressed, these properties are modified by the reduced interatomic distances.

Determining changes in molecules that are subjected to extreme pressure has been made possible by an apparatus containing two carefully ground diamonds with their flat surfaces perpendicular to a beam of light. Between these surfaces is placed a specimen to be studied. Because diamonds are hard and strong, high pressure can then be applied to the material between them, and the light passing through the diamonds and the specimen can be analyzed with a spectrometer.

The specimen compressed between the diamond surfaces produces a very thin film suitable for infrared study. Microscopic examination insures uniformity of the film on the surfaces of the diamonds. Pressure on the specimen can be calculated by the ratio of load to the area of the diamond surface. In the pressure cell used at the Bureau, the area is approximately 0.00015 in.^{2}. Therefore, with a load of only 70 lb, the pressure in the cell exceeds 400,000 lb/in.^{2}. Because the area is so small, very small quantities of specimen are required. In the laboratory, quantities of only a few tenths of a milligram were needed to study the characteristics of a single compound.

Diamonds are suitable for high-pressure work because of their strength, but not all diamonds are transparent in the infrared range. About 99 percent of all diamonds, known as type I, show numerous absorption bands and are therefore unsuitable for pressure cell windows. The remaining 1 percent, known as type II, appear transparent between 0 and 35 μ, with the exception of one absorption band near 5 μ.

Pressure was found to weaken most of the absorption bands of calcite. The decrease of intensity was greater than one might expect to result simply from the restricted amplitude of oscillations. This greater decrease has not yet been explained.



Device used for applying pressure to solids. As the spring at right is tightened, a lever system transmits pressure to diamond holders which are inserted in circular opening at bottom. It appears that the use of diamonds in this way opens a promising new field of study.

For some materials, the spectrum showed new absorption bands, disappearance of normal bands, splitting of single bands, or combining of double bands.

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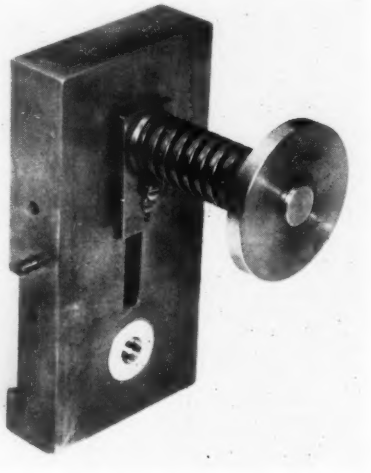

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TECHNICAL NEWS BULLETIN

U.S. DEPARTMENT OF COMMERCE

LEWIS L. STRAUSS, *Secretary*

NATIONAL BUREAU OF STANDARDS

A. V. ASTIN, *Director*

July 1959 **Issued Monthly** **Vol. 43, No. 7**

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. Subscription price, domestic $1.50 a year; 75 cents additional for foreign mailing; single copy, 15 cents. Use of funds for printing this publication approved by the Director of the Bureau of the Budget (June 24, 1958).

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Publications of the National Bureau of Standards

Periodicals

Journal of Research of the National Bureau of Standards, Volume **62**, No. 6, June 1959 (RP2957 to RP2961, incl.) 60 cents. **Beginning with the July issue, the Journal of Research will be issued in four separate sections: A. Physics and Chemistry, annual subscription $4.00, 75 cents additional for foreign mailing; B. Mathematics and Mathematical Physics, annual subscription $2.25, 50 cents additional for foreign mailing; C. Engineering and Instrumentation, annual subscription $2.25, 50 cents additional for foreign mailing; and D. Radio Propagation, annual subscription $4.00, 75 cnts additional for foreign mailing.**

Technical News Bulletin, Volume **43**, No. 6, June 1959. 15 cents. Annual subscription $1.50, 75 cents additional for foreign mailing.

Basic Radio Propagation Predictions for September 1959. Three months in advance. CRPL–D178. Issued June 1959. 10 cents. Annual subscription $1.00, 50 cents additional for foreign mailing.

Research Papers

Journal of Research, Volume **62**, *No. 6, June 1959. 60 cents.*

RP2957. Thermal degradation of polymers at low rates. Samuel L. Madorsky.

RP2958. Pits in metals caused by collision with liquid drops and soft metal spheres. Olive G. Engel.

RP2959. Earth currents near a monopole antenna with symmetrical top loading. James R. Wait.

RP2960. Infrared absorption spectra of some cyclic acetals of sugars. R. Stuart Tipson, Horace S. Isbell, and James E. Stewart.

RP2961. Earth current near a top-loaded monopole antenna with special regard to electrically small L- and T-antennas. H. Lottrup Knudsen.

Index to volume **62** (January to June 1959, Research Papers RP2921 to RP2961).

Circulars

C599. On the theory of fading properties of a fluctuating signal imposed on a constant signal. H. Bremmer. 25 cents.

Building Materials and Structures Reports

BMS152. Wind pressures in various areas of the United States. Guttorm N. Brekke. 15 cents.

Applied Mathematics Series

AMS54. Fractional factorial experiment designs for factors at three levels. W. S. Connor and Marvin Zelen. 30 cents.

AMS56. Tables of osculatory interpolation coefficients. H. E. Salzer. 30 cents.

Publications in Other Journals

Mechanized computation of thermodynamic tables at the National Bureau of Standards: The calculation of the equilibrium composition and thermodynamic properties of dissociated and ionized gaseous systems. Proc. of Symposium—Thermodynamic and Transport Properties of Gases, Liquids, and Solids, Joseph Hilsenrath and Joseph H. Wegstein. Am. Soc. Mech. Engrs., New York, N.Y., 416 (1959).

Nuclear magnetic resonance in semiconductors. Olen Kraus. J. Phys. Chem. Solids **8**, 504 (1959).

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